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EUROPEAN PATENT APPLICATION

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(54) **Crystal growth process for large area GaAs and infrared window/dome made therefrom.**

(57) An optical dome or window formed of a composition which is transmissive to infrared frequencies in the range of from about 1 micron to about 14 microns and which is relatively opaque to substantially all frequencies above about 14 microns consisting essentially of a compound taken from the class consisting of group III-V compounds doped with an element taken from the class consisting of shallow donors and having less than about 1×10^7 atoms/cc impurities and having less than about 1×10^{15} parts carbon. The shallow donors are Se, Te and S, preferably Se, with the Se concentration from 5×10^{15} atoms/cc to 2×10^{16} atoms/cc. The group III-V compound is preferably GaAs or GaP. The group III-V compound is fabricated by providing a graphite vessel (3) containing a graphite cloth (23) with the molten group III element thereover, a shallow donor and water containing boron oxide thereover, loading the group V element into the vessel in essentially stoichiometric amounts to provide the molten compound of the group III and group V elements and cooling the molten compound progressively in a vertical direction from bottom to top to form a crystal by causing growth of the crystal from the bottom up in a vertical direction. The crystal with B_2O_3 thereon is then placed in a vacuum to reboil the B_2O_3 and cause it to foam. The B_2O_3 is then easily removed from the crystal.

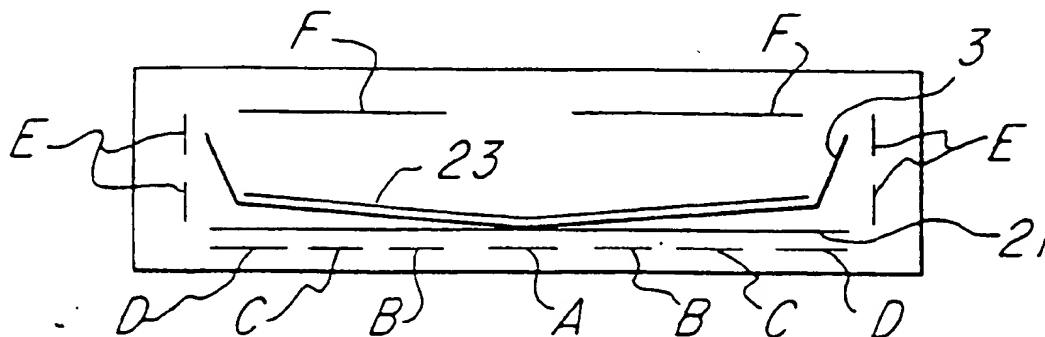


Fig. 2

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EUROPEAN SEARCH REPORT

Application Number
EP 92 30 7647

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.5)
A	PROC. OF THE IEEE 1976 NATIONAL AEROSPACE AND ELECTRONICS CONFERENCE, DAYTON, OH, USA, 18-20 MAY 1976, 1976, NEW YORK, NY, USA page 357 BRAU ET AL 'Development of gallium arsenide for infra red windows' * whole abstract *	1-17	C30B11/00 C30B29/60 G02B1/10 G02B1/02
A	PROC. SPIE EMERGING OPTICAL MATERIALS , SAN DIEGO, CA USA 25-26 AUGUST 1981, vol.297, 1981, SAN DIEGO USA pages 44 - 49 BRAU ET AL 'Gallium Arsenide infrared windows for high speed airborne applications' * whole abstract *	1-17	
A,D	US-A-4 826 266 (BAIRD ET AL)		
A	GB-A-803 347 (SIEMENS)		TECHNICAL FIELDS SEARCHED (Int.Cl.5)
A	PATENT ABSTRACTS OF JAPAN vol. 12, no. 414 (E-677) 2 November 1988 & JP-A-63 152 186 (ITAKUURA) 24 June 1988 * abstract *	1	C30B G02B
A	US-A-4 840 699 (KHATTAK)		
A	EP-A-0 338 411 (ENGELHARD CORPORATION)		
		-/--	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 20 March 1995	Examiner Cook, S
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

EPO FORM 1503 (3.82 (P04C01))



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CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing more than ten claims.

- ☐ All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid, namely claims:
- ☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet -B-

- ☒ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respects of which search fees have been paid, namely claims:
- ☐ None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims,

namely claims:



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EUROPEAN SEARCH REPORT

Application Number
EP 92 30 7647

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.5)
X	JOURNAL OF THE ELECTROCHEMICAL SOCIETY, vol.114, no.9, September 1967, MANCHESTER, NEW HAMPSHIRE US pages 976 - 977 SWIGGARD 'liquid encapsulation zone refining'	18,21,22	
Y	* page 977 *	26	
A	--- PATENT ABSTRACTS OF JAPAN vol. 7, no. 219 (C-188) (1364) 29 September 1983 & JP-A-58 115 085 (FUJITSU K.K.) 8 July 1983 * abstract *	18	
A	--- PATENT ABSTRACTS OF JAPAN vol. 10, no. 90 (C-337) 8 April 1986 & JP-A-60 221 388 (SUMITOMO) 6 November 1985 * abstract *	18	
A	--- JOURNAL OF CRYSTAL GROWTH., vol.30, no.2, 1975, AMSTERDAM NL pages 249 - 256 JOHNSON 'liquid encapsulated floating zone melting of GaAs' * page 252 *	18,22	TECHNICAL FIELDS SEARCHED (Int.Cl.5)
X	--- FR-A-2 345 253 (RTC LA RADIOTECHNIQUE-COMPELEC) * page 4, line 3 - line 10 *	23-25	
X	--- EP-A-0 424 981 (SRI INTERNATIONAL) * page 10, line 56 - page 11, line 25 *	23,24	
A	--- DE-A-34 41 707 (RINGSDORFF-WERKE) * page 7, line 9 - line 31 *	23	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 20 March 1995	Examiner Cook, S
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons dk : member of the same patent family, corresponding document</p>			

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EUROPEAN SEARCH REPORT

Application Number
EP 92 30 7647

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.5)
Y	US-A-4 738 831 (NAUMANN ET AL) * column 4, line 17 - line 38 *	26	
X	DE-A-927 658 (CRESSEL ET AL)	27,28	
Y	* page 3 *	26	
X	PROC. 8TH BIENNIAL CORNELL ELECTRICAL ENGINEERING CONFERENCE, 1981, ITHACA, NEW YORK, US pages 133 - 146 FOOSE ET AL 'growth of undoped semi-insulating GaAs...' * page 135 *	27-29	
			TECHNICAL FIELDS SEARCHED (Int.Cl.5)
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
THE HAGUE		20 March 1995	Cook, S
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

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EP 92 30 7647 -B-

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely:

1. Claims 1-17 : Optical window III-V material and method of preparing it.
2. Claims 18-22 : Method of removing B₂O₃ encapsulant.
3. Claims 23-25 : Method of making compound semiconductor material.
4. Claim 26 : Method of making Si and Ge crystals.
5. Claims 27-29 : Crystalline material having certain resistivity.